

DECLARATION UNDER 37 C.F.R. 1.132

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Sir or Madam:

I, Dr. Franz Simon, hereby declare that:

- 5 1. I, received a M.S. degree in Chemistry from the Johann Wolfgang von Goethe University in 1973 and a Ph.D. degree in Chemistry from Johann Wolfgang von Goethe University in 1976.
2. Over the past 27 years, I have acquired expertise in the area of electrodeposition of glossy gold. In particular, since 1980, I have supervised and performed research on electroplating of precious metals.
- 10 3. I joined UMICORE AG & Co. KG (formerly OMG AG & Co. KG and Degussa) on October 1, 1976 as a leader of a research group where my prime job responsibility has been research and product development of electrodeposition from molten salts, of gold, gold alloys, platinum and silver from aqueous solutions.
- 15 4. I am head of the research and development at UMICORE Galvanotechnik GmbH, a subsidiary of UMICORE AG & Co. KG, and as such responsible for all patents and patent applications of this branch of UMICORE.
- 20 5. I submit this declaration based upon; (i) my training, knowledge, education, and experience as a chemist; (ii) my review of the above-referenced application and the history of the prosecution of this application as reflected in the file maintained by the attorneys prosecuting this application; (iii) my review of prior art cited by the Examiner during prosecution of this application; and (iv) my review of results obtained from further experiments on high speed deposition with an electrodeposition bath according to the claimed invention and in comparison with an electrodeposition bath not according to the claimed invention. The results of these experiments are given in EXHIBIT A.
- 25 6. In EXHIBIT B further examples are given with other combinations of the first and second gloss additive. These additional examples also fully confirm the claimed invention.

7. The present claims are directed to an electrodeposition bath containing at least one specific gloss additive selected from the group consisting of pyridine-3-sulfonic acid, nicotinic acid, nicotinic acid amide, 3-(3-pyridyl)-acrylic acid, 3-(4-imidazolyl)-acrylic acid, 3-pyridylhydroxymethanesulfonic acid, pyridine, 5 picoline, quinolinesulfonic acid, 3-aminopyridine, 2,3-diaminopyridine, 2,3-di-(2-pyridyl)-pyrazine, 2-(pyridyl)-4-ethanesulfonic acid, 1-(3-sulfopropyl)-pyridinium betaine, 1-(3-sulfopropyl)-isoquinolinium betaine or salts or derivatives thereof in combination with a sulfonate or sulfate of the general formula I as an additional gloss additive.
- 10 8. The invention as claimed lies in the combination of a first and, an additional gloss additive selected from two distinct groups of compounds. E.g. the first gloss additive pyridine-3-sulfonic acid does not belong to the second group of gloss additives according to the general formula I since formula I specifies that 15 aryl and or heteroaryl groups (and hence pyridine) are only allowed in combination with $m = 4$, that is only when the additional gloss additive is a sulfate and not a sulfonic acid.
9. In one embodiment of the present invention, the electrodeposition bath of the claimed invention achieves improved current density/working range and improved rate of deposition. Example 1 shows the working range can be up to 20 A/dm^2 if pyridine-3-sulfonic acid is used alone as a gloss additive which corresponds to a deposition rate of $0.98 \mu m/min$ (pages 7 to 8, lines 23 to 31 and 1 to 15). The addition of 1 g/l of nonyl sulfate as a further gloss additive increases the maximum current density to more than $5 A/dm^2$. This corresponds to an improvement of more than 66 %. These results were unexpected.
- 25 10. Examples 2 to 6 of the above-identified application further illustrate the improved results on the working range and the deposition rate achieved by combining the gloss additive and additional gloss additive. Table 1 on page 11 shows the improved deposition rate and working range when both the gloss additive and additional gloss additive are combined. These results were also un- 30 expected.
11. With respect to the CH Patent No. 629,258 ("Marka") and US Patent No. 3,929,595 ("Biberbach"), cited by the Examiner in the Final Office Action of September 4, 2003, I disagree with the Examiner that it is well within the ordi-

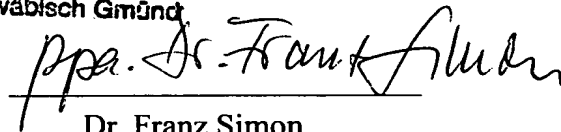
nary skill of the artisan to expect a synergistic effect when using two gloss additives or brighteners in the electrodeposition bath.

12. Marka discloses an acid bath for plating a white gold alloy. Marka further discloses that the acid bath contains gold in the form of alkali or ammonium aurocyanide salts, nickel and/or cobalt as water-soluble compounds and at least one free hydroxy- or amino acid or in the form of salts. This bath is characterized in that it further contains at least a sulfur containing surfactant as gloss additive comprising a sulfonic or sulfinic aliphatic and/or aromatic organic compound. The bath is further characterized in that the hydroxy- or amino acid comprises 2 to 8 carbon atoms, an NH_2 -group or 1 to 6 OH-groups and 1 to 3 COOH groups, whereby the acid functions of these compounds are free or replaced with alkali metal or ammonium (see claim 1 of Marka). Marka does not disclose the specific gloss additive in combination with a sulfonate or sulfate of the general formula I as an additional gloss additive.
13. The Examiner refers to col 2, lines 6 to 8 of Biberbach in the Final Office Action where it is stated that the addition of pyridine-3-sulfonic acid to a gold plating bath allows the bath to be operated at higher current yields at higher current densities.
14. Biberbach is in error here. It is well known to the artisan that the addition of such an additive like pyridine-3-sulfonic acid to the plating bath lowers the current efficiency. It is further well known that with increasing current density the current efficiency decreases. Thus, when reading the disclosure of Biberbach the expert could easily recognize that the cited passage of Biberbach is wrong. The expert would not be led to combine Biberbach and Marka.
15. As will be shown by additional measurements done by Umicore Galvanotechnik (see EXHIBIT A), the addition of pyridine-3-sulfonic acid to a plating bath lowers its current efficiency but increases the working range to higher current densities. The current efficiency drops from 48,5 % at the low end of the working range (10 A/cm^2) to only 15,1 % at 70 A/cm^2 in absolute figures.
16. The advantage of adding pyridine-3-sulfonic acid to a plating bath is the fact that the working range is extended to higher current densities. Since the plating speed is proportional to current density times current efficiency there is the chance that the higher current density overcompensates the drop in current ef-

iciency. This is indeed the case for pyridine-3-sulfonic acid. Thus, despite a reduced current efficiency a higher plating speed can be achieved by increasing the current density.

17. The Examiner alleges that it was obvious for the expert to combine the additive of Marka with the additive of Biberbach. This would imply that the expert would be convinced that adding two additives would improve working range and plating speed. But this is not suggested neither by Marka nor by Biberbach. Contrary to the Examiner's position, the expert would have been anxious not to add an additional additive to either Marka or Biberbach since a plating bath is a well balanced system and the addition of a second gloss additive could destroy the advantages achieved with one gloss additive. The attached measurements will show that only the right combination of gloss additives leads to an improvement. Combining two gloss additives from one of the groups cited in our claim 1, e.g. combining pyridine-3-sulfonic acid with nicotinic acid gives a reduction in plating speed.
18. In summary, the results that we obtained were unexpected and are not disclosed in Marka or Biberbach. One of ordinary skill in the art would not look to these references to obtain the specific gloss additive in combination with a sulfonate or sulfate of the general formula I as an additional gloss additive. Moreover, one of ordinary skill would not expect the results we achieved.
19. All statements made herein of my own knowledge are true, and all statements made are based on our expertise and the attached experiments. All statements are made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 10001 of Title 18 of the United States Code.

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February 25, 2004

Date

An acid bath for the electrodeposition of glossy gold and gold alloy layers and a gloss additive for same

Additional Examples

By Uwe Manz

5 **Example 8:** (3-amino pyridine)

A working range of up to 5 A/dm² is produced with a cell current of 2 A in a Hull cell which contains a gold/cobalt electrolyte containing

10 10 g/l gold in the form of potassium gold (I) cyanide
 0,5 g/l cobalt as cobalt sulfate
10 100 g/l citric acid
 1 g/l 3-amino pyridine
 pH adjusted with potassium hydroxide to 4,2

(test-condition: platinized titanium anode; temperature 50 °C, duration 2 min; agitation 500 rpm with a 25 mm magnetic stirring rod). The current efficiency at 5 A/dm² is 18 %
15 and the plating speed is 0,58 µm/min.

By adding 1 g/l of octyl sulfate the maximum current density which can be used is increased to more than 9 A/dm². At 9 A/dm² the current efficiency is 11 %, the plating speed increases to 0,64 µm/min. This corresponds to a widening of the working range by 80 % and an increase in plating speed by 10 %.

20 **Example 9:** (1-(3-sulfopropyl)-pyridine betaine)

A working range of up to 3 A/dm² is produced with a cell current of 2 A in a Hull cell which contains a gold/cobalt electrolyte containing

 10 g/l gold in the form of potassium gold (I) cyanide
 0,5 g/l cobalt as cobalt sulfate
25 100 g/l citric acid
 1 g/l 1-(3-sulfopropyl)-pyridine betaine
 pH adjusted with potassium hydroxide to 4,2

(test-condition: platinized titanium anode; temperature 50 °C, duration 2 min; agitation 500 rpm with a 25 mm magnetic stirring rod). The current efficiency at 3 A/dm² is 44 %
30 and the plating speed is 0,85 µm/min.

By adding 1 g/l of octyl sulfate the maximum current density which can be used is increased to more than 9 A/dm². At 9 A/dm² the current efficiency decreases to 19 % and the plating speed increases to 1,12 µm/min. This corresponds to a widening of the working range by 300 % and an increase in plating speed by 31 %.

5 **Example 10:** (nicotinic acid)

A working range of up to 3 A/dm² is produced with a cell current of 2 A in a Hull cell which contains a gold/cobalt electrolyte containing

	10	g/l gold in the form of potassium gold (I) cyanide
	0,5	g/l cobalt as cobalt sulfate
10	100	g/l citric acid
	1	g/l nicotinic acid
	pH adjusted with potassium hydroxide to 4,2	

(test-condition: platinized titanium anode; temperature 50 °C, duration 2 min; agitation 500 rpm with a 25 mm magnetic stirring rod).

- 15 By adding 1 g/l of octyl sulfate the maximum current density which can be used is increased to more than 9 A/dm². This corresponds to a widening of the working range by 300 %.